SYNOPSIS

Synthesis, Characterization and Antibacterial Study of co-doped Copper Oxide Nanoparticles

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Introduction:-

In last three decade, Nanotechnology is rapidly developing field. The prefix “nano” originates from Greek word “dwarf” which regards to the microscopic size of the particles. It involves designing, synthesis and characterization of material structure by controlling the size and shape of the particles at the nanoscale. Norio Taniguchi in 1974 first time study the semiconductor processes such as thin film deposition and ion beam milling existing characteristic to control the particle size on the range of a nanometer and defined the term nanotechnology. These days, Nanotechnology is the most popular fields of scientific research [1]. The nanosize materials have gained a great attention from the research work not only because of their excellent properties but also due to their various applications [2]. Currently, the focus of researchers is on exploration of metal and metal oxide nanoparticles [3]. Techniques used to characterize the metal oxide nanoparticles are scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), dynamic light scattering (DLS), Fourier transform infrared spectroscopy (FTIR), EDX (Energy-dispersive X-ray spectroscopy), UV-visible spectroscopy [4].

A number of metal oxides are available in the universe but some of the metal oxides are most useful because of their important applications in day to day life [5]. Some of the transition metal oxides like ZnO, CuO, TiO_2, MnO and Fe_3O_4 etc. shows number of applications in different areas [6]. Among them, CuO is a p-type, narrow band gap semiconductor, non-toxic, more stable, insoluble in most of the organic solvents, having monoclinic structure and less expensive antimicrobial agent [7]. CuO nanoparticles have high surface to volume ratio [8], low production cost. These particles show good antibacterial activity, catalytic activity, optical and magnetic properties as compared to other metals such as gold, silver etc. [9].

A number of transition metals have been used for doping, co-doping of pure CuO nanoparticles to change their physical and chemical properties [10]. Doping with metallic atoms on pure CuO nanoparticles can be used to improve the physical properties of pure CuO nanoparticles and the ions doping on the pure CuO nanoparticles can improve the photocatalytic activity of CuO nanoparticles [11]. Recent research work shows that CuO

have variable oxidation states such as Cu^{+}, Cu^{2+}, and Cu^{3+} and this property of CuO nanoparticles give an important role on the development of Cu nanoparticles, because of which hole and electron doping are possible on the pure CuO nanoparticles [3].

CuO nanoparticles can also be used as antimicrobial agent [12]. The antibacterial properties of CuO nanoparticles depends upon their size, stability and concentration added to growth medium, provides greater retention time for bacterium nanoparticles interaction [12]. CuO nanoparticles have antimicrobial properties because they have ability of producing reactive oxygen species (ROS) when they come in contact with water [13]. CuO nanoparticles have attracted most attention because of their antimicrobial activities, biocide properties and they may be used in many biomedical applications [2].

Pure CuO and co-doped CuO nanoparticles have been fabricated by using various physical, chemical and biological methods such as ball milling method, reactive DC sputtering [14], precipitation method [15], sol-gel method [7], spray pyrolysis method [16], sonochemical method [17], microwave assisted method [18], green/biological synthesis method such as fungi, algae, plant and bacteria [2,8,9]. From last few years, a number of chemical synthesis methods are available to synthesize CuO nanoparticles. They can also be synthesized chemically by sol-gel method [7], hydrothermal method [19], sonochemical method [17], microwave irradiation method [18], co-precipitation method [20], simple chemical precipitation method [15] and spray pyrolysis method [16].

Review of Literature:-

Wang H. et al. (2002) has prepared CuO nanoparticles by microwave irradiation method and reported that nanoparticles have monoclinic structure with the particle size 4 nm and band gap 2.43 eV [21]. **Yang S.G. et al. (2003)** has studied ferromagnetism in Mn-doped CuO. They observed ferromagnetic properties in Mn-doped CuO with 3.5-15 % of Mn [22]. **Zhu H.T. et al. (2004)** has studied CuO nanoparticles by sodium hypophosphite reduction in ethylene glycol under microwave irradiation and they concluded that TEM images consists of irregular and strong sinter-like particles with size of about 30-70 nm [23]. **Zhu J. et al. (2004)** reported highly dispersed CuO nanoparticles with an average size of 6 nm

prepared by a novel quick precipitation method [24]. **Li D. et al. (2005)** has prepared CuO nanostructures by chemical method and concluded that characteristic peaks (XRD) and HR-TEM images shows single-crystal core like structure [25]. **Wu C.K. et al. (2006)** has conducted quantitative analysis of copper oxide nanoparticle [26]. **Zhu H.T. et al. (2007)** has studied thermal conductivity of CuO nanofluid. They concluded that microwave irradiation method may be used for CuO nanofluid synthesis which shows needle-like particles, single shuttle-like particles with a particle size in the range of 30-90 nm [27].

Khanna P.K. et al. (2007) has studied synthesis and characterization of copper nanoparicles. They observed that freshly prepared Cu nanoparticles solution have an absorption band at about 600 nm due to surface plasmon resonance (SPR) [28]. **Ruparelia J.P. et al. (2008)** has studied antimicrobial activity of silver and copper nanoparticles and reported that the average size of the silver and copper nanoparticles is 3 nm and 9 nm respectively [29]. **Meneses C.T. et al. (2008)** has studied synthesis and characterization of Transition Metal (TM) doped CuO (TM = Fe, Ni). They observed single-phase structure by XPRD [20]. **Son D.I. et al. (2009)** has studied optical and electronic properties of colloidal CuO nanoparticles formed by colloid-thermal synthesis process and observed monoclinic structure with optical band gap energy is 3.63 eV [30]. **Pereishtein I. et al. (2009)** has prepared CuO nanoparticles of size 15 nm by using ultrasound irradiation method [31]. **Ren G. et al. (2009)** has studied characterization of CuO nanoparticles for antimicrobial applications and TEM pattern reported particle size in the range 20-95 nm [32].

Gao W. et al. (2010) has studied synthesis and magnetic properties of Mn-doped CuO nanowires. TEM study indicated a single crystal property of the nanowires [33]. **Li Y. et al. (2010)** has studied ferromagnetic properties of Fe-doped CuO nanocrystals. XRD analysis confirmed the monoclinic structure of CuO [34]. **Joseph P. et al. (2010)** has studied magnetic properties of Nanocrystalline Cu-Fe-O by co-precipitation and ceramic methods [35]. **Haseini A. et al. (2011)** has studied fabrication of CuO: Fe nanoparticles. The TEM Patterns shows average diameter of nanoparticles was 37 nm [36]. **Jing Z. et al. (2012)** has studied ferromagnetism in Fe-doped CuO nanopowder and XRD spectra confirmed the monoclinic structure of CuO [37]. **Mohebbi S. et al. (2013)** has investigated Sn-doped CuO nanoparticles prepared by hydrothermal method and X-ray analysis result revealed that average particle size of nanoparticles increases from16 nm to 22 nm [19]. **Rejitha S.G. et**

al. (2013) has studied the cadmium doped CuO nanoparticles and recorded particle size for pure CuO nanoparticles is 10-14 nm and for doped CuO nanoparticles 42-87 nm [18].

Basith N.M. et al. (2013) has studied Fe-doped CuO nanostructures and reported single-phase monoclinic nanostructure with optical band gap 2.8 eV for pure CuO calculated by using DRS and increases up to 3.4 eV with increasing 'Fe' content [38]. **Gulen Y. et al. (2013)** has investigated Mn-doped CuO thin films and found that all the films have monoclinic structure and optical band gap enhance with the increasing of doping concentrations [39]. **Jan T. et al. (2014)** has studied antibacterial properties of Ce-doped CuO nanomaterial with an average diameter of 30 nm [40]. **Malka E. et al. (2014)** has prepared Zn-doped CuO nanocomposite of size 30 nm and study their antibacterial properties against E. coli and S. aureus [17]. **Baisth M. et al. (2014)** has studied Co-doped CuO nanostructures. They reported monoclinic structure with band gap 2.1 eV for pure CuO and raise up to 3.4 eV with increasing concentration [41]. **Viruthagiri G. et al. (2014)** has reported that ZrO_2-CuO co-doped ceria nanoparticles of size ranges from 5-6 nm and Photoluminous (PL) spectra confirmed strong and sharp UV emission at 401 nm [42].

Eshed M. et al. (2014) has prepared Zn-doped CuO nanocomposite by sonochemical method. They reported average particle diameter 30-70 nm and nanoparticles formed illustrate antibacterial activity against S. multans [43]. **Ahamed M. et al. (2014)** has studied antimicrobial activity of CuO nanoparticles [44]. **Radhkrishnan A. et al. (2014)** has informed about antimicrobial properties of CuO nanoparticles of size 19 nm against gram-positive and gram-negative bacterial strains [45]. **Bayansal F. et al. (2014)** has studied effect of Co-doping on CuO thin films and SEM analysis confirmed the grain size of nanostructures as 70, 44, 42, 36, 32 and 38 nm with increasing doping concentration [46]. **Dhanuskodi S. et al. (2014)** has investigated dielectric properties of Fe-doped CuO nanoparticles [47]. **Ramya S. et al. (2015)** has prepared Ni^{2+} ions incorporated CuO nanoparticles by precipitation method and confirmed its antibacterial activity against B. subtilis and E. coli [5].

Fei Z.J. et al. (2015) has investigated monoclinic phase of Fe-doped CuO nanoparticles prepared by hydrothermal method [11]. **Sharma N. et al. (2015)** has confirmed size of Mn-doped CuO nanostructures as 116, 75, 55, 40 nm with increasing dopant content [48].

Hemalatha T. et al (2015) has prepared zinc-doped Copper oxide Nanoparticles of size 18-25 nm [15]. **Cai X.M. et al. (2015**) has studied the indium-doped Cu_2O thin films [14]. **Sayed A.M. EI et al. (2015)** has studied Fe and Co, Fe co-doped CuO nanoparticles and concluded that the average crystallite size of pure CuO is 20.44 nm, which reduced to 18.72 nm after Fe doping, then increased to 26.82 nm due to co-doping with Co atoms [49]. **Mishjil K.A. et al. (2015)** has prepared Mn-doped CuO nanoparticles by spray pyrolysis technique and recorded their optical properties [16]. **Al-Amri S. et al. (2015)** has prepared Ni doped CuO nanoparticles of size 23-47 nm via sol-gel method [50]. **War N.G.T. et al. (2016)** has synthesized CuO nanoparticles by microwave assisted method. XRD pattern confirmed nanoparticles of size 23 nm [51]. **Khmissi H. et al. (2016)** has employed sol-gel spin coating technique to prepare Ni and La, Ni co-doped CuO nanoparticles and recorded particle size is 14.23-28.32 nm [52].

Yakout S.M. et al. (2016) has investigated monoclinic structure of pure and (Mn, Co) co-doped CuO nanostructures [53]. **Kadhima R.G. et al. (2016)** has studied pure and Li-doped CuO nanoparticles and concluded that size increases with concentration of Li [7]. **Kavitha K. et al. (2016)** has prepared Cu doped tin oxide and tin doped CuO thin films by sol-gel method [54]. **Sasikala R. et al. (2016)** has studied antibacterial properties of Lanthanum, Cerium and Erbium loaded CuO nanoparticles [12]. **Jayasree K.V. et al. (2016)** has investigated Cr doped CuO nanoparticles and concluded that average grain size of doped nanoparticles varies from 22.14 nm-17.5 nm [55]. **Rani P. et al. (2016)** has conducted study on Fe doped CuO nanoparticles and reported particles size lies is in range 8-35 nm [56]. **Kannaki K. et al. (2016)** has reported Fe doped CuO nanocomposites prepared by hydrothermal method [57]. **Varghese R. et al. (2017)** has prepared Mg-doped CuO nanoparticles by quick precipitation method [58]. **Iqbal M. et al. (2017)** has studied Mn-doped CuO nanowires and reported monoclinic structure [10].

Research Gap Identification:-

A number of research groups have reported doping of transition metals in pure CuO nanoparticles and very small research literatures is available on co-doping in pure CuO

nanoparticles. It is also found from literature survey that less research work has been done on co-doped CuO nanoparticles and their antimicrobial/antibacterial properties.

The current research work is devoted to the study of pure and co-doped CuO nanoparticles, which have been fabricated by using the microwave assisted method.

Objectives:-

1. To synthesize pure and co-doped CuO nanoparticles using microwave assisted method.
2. To characterize synthesized nanoparticles.
3. To examine the antibacterial properties of synthesized co-doped CuO nanoparticles.

Proposed Methodology:-

Chemicals:-

Copper (II) acetate monohydrate, cobalt (II) acetate tetrahydrate, zinc acetate dihydrate, silver nitrate, polyethylene glycol (PEG), KOH, nickel (II) chloride hexahydrate, magnesium chloride, iron (II) chloride tetrahydrate, aluminium chloride anhydrous purchased from merck.

Media: -Nutrient broth, Nutrient agar and Mueller Hinton broth, Mueller Hinton agar.

Synthesis Procedure:-

Pure CuO Nanoparticles:-

Pure CuO nanoparticles will be synthesized by using microwave assisted method. For the synthesis of pure CuO nanoparticles, copper (II) acetate monohydrate and potassium hydroxide pellets shall be used as precursors. Polyethylene glycol shall be used as stabilizer and capping agent. Dissolve copper (II) acetate monohydrate in 100 ml of distilled water and magnetically stirrer without heating for 15-20 minutes. Dissolve PEG in 100 ml of distilled water, added drop-wise to the above prepared solution which is continuously magnetically stirrer. Then KOH pellets will be dissolved in 100 ml of distilled water, add slowly into the above solution and magnetically stirrer solution of copper (II) acetate monohydrate, PEG and KOH solution for an hour, until pH reached 9. The obtained solution will be heated in a

microwave oven with operating frequency 2.45 GHz and power 800 W for few minutes. Microwave irradiation completely evaporates the solvent. After the microwave processing, the prepared solution will be cooled at room temperature. Centrifuge the obtained precipitate for 15 minutes, then washed with distilled water and ethanol for several times and finally obtained precipitate will be dried in an oven at 60^0C for 24 h.

co-doped CuO Nanoparticles:-

co-doped CuO nanoparticles will be synthesized by using microwave assisted method. For synthesis of co-doped CuO nanoparticles, copper (II) acetate monohydrate and potassium hydroxide (KOH) pellets will be used as precursors. Cobalt (II) acetate tetrahydrate, silver nitrate, nickel (II) chloride hexahydrate, zinc acetate dihydrate, magnesium chloride, iron (II) chloride tetrahydrate, aluminium chloride anhydrous etc. shall be used for co-doping material of CuO nanoparticles. Appropriate amounts of copper (II) acetate monohydrate will be dissolved in 100 ml of distilled water. For co-doping the solution of two metal salts will be added drop-wise combinedly to the above prepared solution with constant stirring. KOH pellets dissolved in 100 ml of distilled water will be added slowly into the above solution, until the pH reached 9. Dissolved PEG in 100 ml of distilled water will be added drop-wise to the above solution. The obtained solution will be kept in the microwave oven with operating frequency 2.45 GHz and power 800 W. The microwave irradiation will be resulted in the black color solution. After the microwave processing, the nanoparticles solution will be cooled at room temperature and nanoparticles washed with distilled water and ethanol, several times and finally nanoparticles precipitate will be dry in an oven at 60^0C for 24 h. The synthesized sample will be characterized by using various techniques and antibacterial activity test will be performed for the nanoparticles.

Antibacterial Activity:-

Antibacterial activites of pure and co-doped CuO nanoparticles will be tested with the help of well diffusion method by using Staphylococcus aureus (MTCC 737), Bacillus subtilis (MTCC 441), Pseudomonas aeruginosa (MTCC 1688), Escherichia coli (MTCC 739). Test organisms (Bacterial strains) will be collected from Institute of microbial Technology, microbial Type culture collection centre, (IMTECH), Chandigarh. Mueller-

Hinton agar will be poured in the sterilized petri plates and allowed to solidify. The plates will be left over night at room temperature to check for any contamination to appear. Bacterial strains will be spreaded on Mueller-Hinton agar plates and produced the hole with the opposite side of tip. The prepared CuO and co-doped CuO nanoparticles suspension will be added into the wells. These samples initially incubated at 37^0C for 24 h. In addition, the minimum inhibitory concentration (MIC) inhibits the growth of organisms, can be determined.

Thesis Outline:-

The arrangement of the thesis in the forms of chapters will be as follows:

Chapter 1:- This chapter will contain the complete introduction.

Chapter 2:- This chapter will contain the review of literature.

Chapter 3:- This chapter will give the methods for synthesis of pure and co-doped CuO nanoparticles and their antibacterial study.

Chapter 4:- This chapter will contain detail calculations and results obtained from the synthesis and characterization of pure and co-doped CuO nanoparticles.

Chapter 5:- This chapter will include the possible and even critical discussion on the results obtained in the present investigation.

Chapter 6:- This chapter will present the conclusion that could be drawn out of the present research work.

Chapter 7:- This chapter will include the references related to the study.

Research Plan Schedule:-

Research Activity	Progress in Month											
	3	6	9	12	15	18	21	24	27	30	33	36
Course work, extensive literature survey or design of work	Aug 2016 to July 2017											
Research gap identification and development of methodology		July 2017 to Dec 2017										
Experimental Work or Field Work					Dec 2017 toJun 2018							
Lab Work and Calculations						Jun 2018 to dec 2018						
Conclusion, manuscript preparation for publication and thesis writing								Dec 2018 to July 2019				

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